

## SHEAR BOND STRENGTH OF ORTHODONTIC METAL BRACKETS BONDED TO TWO DIFFERENT TYPES OF ZIRCONIUM CROWNS: AN IN-VITRO COMPARATIVE STUDY

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### ABSTRACT

**Objectives:** Adults want aesthetic restorations like monolithic zirconia crowns. Due to the unique surface treatment, orthodontists have trouble bonding orthodontic braces to this material. This study examines the shear bond strength (SBS) of metal brackets attached to two zirconia ceramics, surface roughness (SR) following hydrofluoric acid surface treatment, and adhesive residual index (ARI). **Materials and Methods:** Twenty crowns of monolithic zirconia and twenty crowns of monolithic high transparent zirconia (Zolid-HT/W) were fabricated. The surface was treated with hydrofluoric acid etching. Twenty extracted lower central incisors were prepared and etched with hydrofluoric acid (HF) to serve as the control group. SR, SBS, and ARI were evaluated. **Results:** Enamel and Zolid-HT/W groups had the greatest SBS and SR values, respectively. **Conclusion:** With HF surface treatment, Zolid-HT/W and zirconia groups may achieve sufficient bond strength. **Clinical significance:** To get the best findings about the adhesive strength of orthodontic brackets, a portion of a simulation was conducted in a manner analogous to clinical practice.

**KEYWORDS:** Shear bond strength. Zirconium crowns. Surface treatment. Orthodontic brackets.

### INTRODUCTION

Today, patients desire aesthetic crowns made entirely of ceramic or fixed partial dentures that match their demands. Zirconium oxide (ZrO<sub>2</sub>) is a material created by dental manufacturers that offers various advantages over standard porcelain

fused to metal (PFM) prostheses, including a more elegant appearance, increased chemical properties, and greater mechanical qualities.<sup>[1, 2]</sup> Nevertheless, veneering porcelain chipping is greater with zirconia cores than PFM. Fired porcelain shrinks and the core and porcelain's thermal expansion coefficients differ, causing porcelain chipping.<sup>[3, 4]</sup>

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On the basis of the above, monolithic zirconia is regarded as the remedy for porcelain chipping<sup>[3,5]</sup> Bonding on monolithic zirconia is a dilemma for orthodontists because to the increased bonding failure rate of brackets applied to these surfaces compared to enamel.<sup>[6, 7]</sup> Etching with hydrofluoric acid cannot increase bond strength,<sup>[3, 7]</sup> as zirconia does not have a glass phase,<sup>[8,9]</sup> Yet, it may increase the binding strength of new monolithic zirconia manufactured by companies that added additional oxides to the zirconia's composition to improve its appearance. Silanization has also been suggested to improve bonding<sup>[10,11]</sup>.

With the constant evolution of zirconia to new versions for aesthetic enhancement, it becomes vital to evaluate the SBS of orthodontic brackets on these new versions in comparison to older zirconia with different surface pre-treatments. This may be useful for researching the effect of new monolithic zirconia versions on the bonding strength of brackets.

This study examines how hydrofluoric acid etching affects metal brackets' shear bond strength to monolithic zirconia crowns. After failure, the crowns' remnant cement was examined.

## MATERIAL AND METHODS

### Sample

The study was authorized by Mansoura University's ethics committee. The inclusion criteria were as follows: (1) crowns with no major flaws, restorations, or crack lines; (2) no chemical agents, such as formalin or hydrogen peroxide treatment; and (3) labial and lingual surfaces had never been glued to orthodontic attachments, and (4) teeth had normal clinical size based on crown height and width estimations. These removed lower central incisors were utilized as a comparative group.

Twenty monolithic zirconia crowns (group I, n=20) and twenty monolithic high transparent zirconia crowns (group II, n=20) were fabricated. Twenty extracted central incisors from the lower jaw (group III) were prepared.

The sample size was calculated by using G\*Power software (version 3.1.9.7). Based on previous studies,<sup>[11, 12]</sup> we hypothesized large effect size ( $f=0.5$ ) when comparing the three groups as regards both SBS and surface roughness.

In a one-way ANOVA study, sample sizes of 20 Enamel, 20 Zirconia, and 20 Zolid-HT/White are obtained from the 3 groups whose means are to be compared. The total sample of 60 subjects achieves 93% power to detect differences among the means versus the alternative of equal means using an F test with a 0.0500 significance level. The size of the variation in the means is represented by the effect size  $f = \sigma_m / \sigma$ , which is 0.5000.

### Procedures

After cleaning, the lower central incisors were stored in a physiological saline solution at 5°C for 90 days before testing. Their crowns were scanned using an extra-oral 3D scanner (DOF Inc., ASD 180323002Q, Korea) to create labial surfaces for the two types of monolithic zirconia crowns using CAD (Exocad DentalCAD Matera 2.3 program). This made glueing brackets on labial surfaces easier. (Figure 1).

The monolithic high transparent zirconia crowns (Ceramill ® Zolid ht+ White) and Natura Eco HT (DMAX Co. Ltd., Korea) were milled by CORiTEC 250i, a CAM machine (imes-icore GmbH, Im Leibolzgraben 16 D-3132 Eiterfeld, Germany). All milled crowns were sintered in a specific furnace ZIRKON-100 (MIHM-VOGT GmbH & Co. KG, Friedrich-List-StraBe 8, 76297 Stutensee-Blankenloch, Germany) under 1450°C for 8 hours, then glazed in a glazing machine Programat P310 (Ivoclar Vivadent AG, FL-9494 Schaan, Liechtenstein, Austria) under.<sup>[13]</sup>

In groups, I and II, a proprietary gel base Hydrofluoric acid 9.6% (HFA) (BISCO, Inc., 1 W. Irving Park Rd., Schaumburg, Illinois 60193, USA) was utilized for chemical pretreatment for 30 seconds<sup>[14, 15]</sup>. The crowns were air-dried for 30

seconds after washing under tap water for almost half a minute. In group III, all labial surfaces were etched with 37% phosphoric acid (PHA) in a proprietary gel base (Meta Biomed Co., Ltd. 414-12 Mochung-Dong, Heungdeok-gu, Chungbuk, Korea) for 20 seconds, rinsed under tap water for 30 seconds, and gently air-dried till chalky white.

Before silanization or bonding, surface roughness was determined by profilometer (SURFTTEST SJ-201, Mitutoyo Corp., Japan) for all groups. The stylus traversed the specimens. Five measurements yielded the average roughness value (Ra). At 0.5 mm/sec scanning speed, 0.8 mm was the cut-off length. Scanning quality was 0.01 µm.

Groups I and II's labial surfaces were primed with a thin coating of RelyX Ceramic Primer (3M ESPE, St. Paul, US) for 20 seconds with an application brush and air-dried with a mild, oil-free air spray. For all groups, a micro-brush applied a thin coating of universal bond, Transbond™ XT primer (3M Unitek, Monrovia, California, U.S.), then gently air-thinned. As primer is photopolymerizable, intense ambient light was avoided.

Metal bracket bases were coated with enough Transbond™ XT paste (3M Unitek, Monrovia, California, U.S.). After softly placing the metal brackets on the crowns, a bracket positioner was used to standardize the technique, and the brackets

were firmly pushed down. Removing excess cement was done. Last, brackets were cured for 40 s using a 1,200 mW/cm<sup>2</sup> curing light (C02-C LED Premium Plus International Ltd. 1001, Yuen Long Trade Centre No.33, Wang Yip Street West Yuen Long, N.T. Hong Kong) (10 s from mesial, 10 s from incisal, 10 s from distal, and 10 s from gingival directions). Epoxy resin blocks contained samples from the root.

After bonding, each sample underwent 24 hours of incubation at 37°C. They were subjected to 1,000 heat cycles at 5-5°C (± 4°C) in distilled water baths, with a 20-second interval between baths [11,16,17]. The thermocycler was used for this (Robota, Alex, Egypt). Each specimen was positioned in the Instron universal testing machine such that it touched the incisal wings of every bracket. Such a location was necessary to achieve the maximal shear stress parallel to the crown surface at 1 mm/min cross-head speed. (Figure 1).

SBS was calculated in MPa using the formula  $Mpa = F/A$ , where F is the highest load recorded in N by the Instron machine after debonding and A is the surface area of the metal bracket base in mm<sup>2</sup> (9.4 mm<sup>2</sup>)<sup>[18]</sup>. Following failure, 3.5x loupes determined the ARI.<sup>[19]</sup> Cohesion, adhesion, or mixed described each sample.

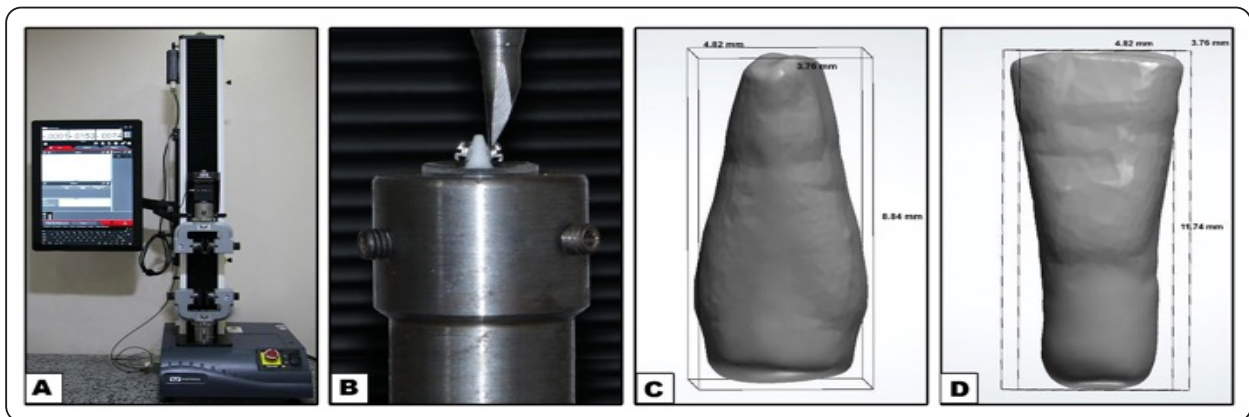


Fig. (1): (A) Instron machine (B) To maximise parallel force from blade to buccal crown surface, samples were kept in epoxy resin blocks. (C) Bucco-lingual view of milled zirconium crowns. (D) Mesio-distal view milled zirconium crowns.

**STATISTICAL ANALYSIS**

Data were entered and analyzed using IBM-SPSS software (Version 26.0). Qualitative data were expressed as N (%) and compared by using the Fisher-Freeman-Halton test followed by multiple 2 X 2 Fisher’s exact tests with adjusted p-value of 0.0167 for the three comparisons using Bonferroni method of correction. Quantitative data were initially tested for normality using Shapiro-Wilk’s test, and data were considered as normally distributed (Shapiro  $p > 0.050$ ). The presence of significant outliers was tested for by inspecting boxplots. Quantitative data were expressed as mean  $\pm$  SD and compared between the three groups by one-way ANOVA-test. As homogeneity of variances was violated (Levene’s test  $p < 0.05$ ), Welch ANOVA and Games-Howell post hoc tests were reported.

**RESULTS**

The three groups shows a statistically significant difference between them as regards both SBS

and surface roughness. Post hoc tests revealed a statistically significantly lower SBS in Zirconia group vs. each of the two other groups but not between Enamel and Zolid-HT/W groups, and a statistically significantly lower surface roughness in Zirconia group < Zolid-HT/W group < Enamel group. (Table 1).

The three groups also shows a statistically significant difference between them as regards ARI. For this statistically significant Fisher’s exact test (2 x c), multiple Fisher’s exact tests (2 x 2) were performed to determine which of the three groups differ. For the result to be statistically significant,  $\alpha$ -level was adjusted to a significant level of 0.0167 by using Bonferroni correction. ARI was not statistically significantly different between Enamel and Zirconia group ( $p = 0.741$ ), statistically significantly higher in Zolid-HT/W group vs. enamel group ( $p = 0.003$ ), and marginally significantly higher in Zolid-HT/W group vs. Zirconia group ( $p = 0.020$ ) (Table 2)

TABLE (1): SBS and surface roughness in the three groups

Measure	Group			One-way Welsh ANOVA			Post hoc Games-Howell		
	Enamel	Zirconia	Zolid-HT/W	F	p-value	$h^2$	P1	P2	P3
SBS	32.6 $\pm$ 14	15.03 $\pm$ 2.9	29.7 $\pm$ 8.3	39.281	<b>&lt;0.001</b>	0.405	<b>&lt;0.001</b>	0.719	<b>&lt;0.001</b>
Surface roughness	1.058 $\pm$ 0.07	0.241 $\pm$ 0.04	0.401 $\pm$ 0.15	993.405	<b>&lt;0.001</b>	0.934	<b>&lt;0.001</b>	<b>&lt;0.001</b>	<b>&lt;0.001</b>

Notes: Data: Mean  $\pm$  SD. SBS = Shear bond strength. HT/W = HT/White.  $h^2$  = effect size measure. P1 = Enamel vs. Zirconia, P2 = Enamel vs. Zolid-HT/White, and P3 = Zirconia vs. vs. Zolid-HT/White.

TABLE (2): ARI in the three groups

ARI	Group						Total		p-value
	Enamel		Zirconia		Zolid-HT/White		N	%	
	N	%	N	%	N	%			
Cohesive	12	60.0%	14	70.0%	20	100.0%	46	76.7%	<b>0.005</b>
Adhesive	8	40.0%	6	30.0%	0	0.0%	14	23.3%	
total	20	100.0%	20	100.0%	20	100.0%	60	100.0%	

Notes: The test of significance is Fisher-Freeman-Halton Exact Test.

## DISCUSSION

This study compares the shear strength of metal orthodontic brackets attached to two types of CAD/CAM monolithic zirconia crowns with Hydrofluoric acid 9.6% surface treatment to the enamel control group. It was also examined how zirconia crown types affected orthodontic bracket shear bond strength.

For glueing brackets to zirconia with the proper SBS, zirconia must be prepared by a variety of mechanical or chemical techniques, such as strong acids, before being bonded to the brackets. Prior research demonstrated that the use of HF acid as an etchant does not result in an adequate binding strength[5, 20]. HF etching produced significant surface roughness for the (Zolid ht+ white) group in the present investigation. On the second sort of zirconia, however, HF had no impact. The variation in HF's impact on the two kinds of zirconia may be attributable to their distinct chemical compositions and production processes..

Many research featured in a systematic review studied the effects of varying concentrations of hydrofluoric acid on zirconia samples as an etchant (9.6%, 5%, and 4% HF).<sup>[11, 21-24]</sup> The theory of using HF acid to etch the surface of the crowns we provided is that (Zolid ht+ white) is not a noble zirconia crown since it was changed with various oxides ( $ZrO_2 + HfO_2 + Y_2O_3: \geq 99.0$ ,  $Y_2O_3: 6,7 - 7,2$ ,  $HfO_2: \leq 5$ ,  $Al_2O_3: \leq 0.5$ , Other oxides:  $\leq 1$ ) by the manufacturer, which may be the reason of its increased translucency and high strength compared to the other kind.<sup>[25]</sup> Hydrofluoric acid etching affects the micro-morphology of glassy ceramics depending on their chemical composition and microstructure organization.<sup>[26]</sup>

Material properties of Feldspathic VITA Mark II possesses the exact same and cumulative percentage of constituents as leucite-reinforced IPS Empress CAD, lithium disilicate IPS e.max CAD, and zirconia-reinforced lithium silicate

Celtra Duo, but the different chemical distribution creates various etching behaviours on the surfaces. According to supplier records, the ceramics' silica and alumina composition is:  $SiO_2$ : 56%, 60%, 80%, 56%;  $Al_2O_3$ : 20-23%, 16%, 5%, and 4%. All glassy ceramics include 60% silicon dioxide, whereas feldspar-based and leucite-reinforced ceramics have 20% aluminium oxide, and lithium-disilicate and zirconia-reinforced lithium silicate contain 4%. HF acid etching is affected by the physical structure and other oxides.<sup>[26]</sup>

A recent integrative study found that most writers employed solely LED photo activators, making Reynolds in 1975 estimates shear bond strength results were not accurate. After eleven tests, the clinically acceptable SBS was  $16.14 \pm 11.13$  MPa without enamel damage.<sup>[27][28]</sup>

Kwak, J.-Y., et al. advocated a silane primer for zirconia with a porcelain glaze, but a zirconia primer for the exposed surface.<sup>[11]</sup> Our SBS results are clinically satisfactory for most zirconium groups, such Kwak, J.-Y., et al.

In our study, the control enamel group showed higher SBS mean values than other groups. This findings mean that increasing the surface roughness may enhance the shear bond strength.

Young's modulus of Zolid HT/ W is 200 GPa, and its bending strength is 1100–150 MPa. Debonding the bracket from the crown surfaces is unlikely to cause fractures. The connection between the prosthetic crowns and the tooth depends on several factors, including the cementation utilized. The crown bonded to the bracket must be in balance to prevent debonding. Zirconia crowns' top strength limit is unknown.<sup>[29]</sup>

Debonding a bracket requires preserving the enamel or zirconia surface's structure and leaving as little adhesive residue as feasible. Low ARI scores prevent zirconia coherent breakdown.<sup>[30]</sup> In this study, the Zolid HT/W displayed the lowest mean ARI score.



Thermo-cycling exposes an extracted tooth with a restoration to oral cavity-like temperatures *in vitro*. First, temperature-induced mechanical stresses may directly create fractures across bonded surfaces. Second, pathogenic oral fluids in and out of gaps cause variable gap widths. Material linear coefficient of thermal expansion affects micro-leakage.<sup>[31]</sup> As thermo-cycling represents the worst-case situation for ageing. Data extraction was limited to non-thermo-cycled and thermo-cycled groups, per a systematic review and meta-analysis<sup>[32]</sup>. In addition, the number of cycles varied widely, ranging from 500 to 37,500. Hence, the provided data revealed substantial standard deviations. In general, thermocycling seems to impair bond strength, therefore 1000 cycles were used for this investigation since they were within the range cited in a prior systematic review. In addition, the thermocycler accessible at our institution has a maximum range of 1,000 cycles each day.

The occurrence of cycling *in vivo* is now unknown and needs proper evaluation. In the absence of this information and based on the assumption that these cycles might happen between 20 and 50 times each day, it is hypothesized that one year of *in vivo* behavior may consist of 10,000 cycles.<sup>[33,34]</sup> No data exist about the number of heat cycles per unit time *in vivo*, which obviously calls for more study.

## CONCLUSION

Adequate bond strength could be obtained with Zolid-HT/W and zirconia group if bonded with metal brackets after HF surface treatment.

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## Declaration of interest's statement

The authors declare no conflict of interest.

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